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TECHNICAL NOTE

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HIGH-TEMPERATURE TENSILE PROPERTIES OF

WROUGHT SINTERED TUNGSTEN

By Paul F. Sikora and Robert W. Hall

Lewis Research Center Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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TECHNICAL NOTE D-79

HIGH-TEMPERATURE TENSILE PROPERTIES OF

WROUGHT SINTERED TUNGSTEN

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SUMMARY

Specimens from 1/2-inch-diameter, commercially pure, sintered, and swaged bars supplied by five producers were evaluated in short-time tensile tests at temperatures from 2500° to 4400° F. Results showed that the decrease in ductility of tungsten at high temperatures determined in a previous investigation is characteristic of commercially pure sintered tungsten. However, an appreciable amount of ductility is still retained up to 4400° F. The tensile strength of tungsten decreased from about 10,600 at 3500° to 4400 psi at 4400° F.

INTRODUCTION

The refractory metals are becoming increasingly important as potential structural materials in high-speed aircraft, missiles, and space vehicles. Tungsten, in particular, is being considered for these applications because of its high melting point (6170°F) and relatively high strength at temperatures above 3000° F. However, the available information on the mechanical properties of tungsten above 2500° F is very limited. In a previous investigation (ref. 1), the short-time tensile properties of commercially pure tungsten from one commercial supplier were determined in the temperature range of 2500° to 3700° F. One purpose of the present study was to extend the data to a higher temperature, 4400° F. In the previous study, it was observed that tungsten exhibited a decrease in ductility above about 3100° F. A second purpose of the investigation reported herein was to determine whether this decrease in ductility at high temperatures was generally characteristic of commercially pure tungsten or only of the one lot of material previously evaluated. Commercially pure tungsten bars supplied by five different producers were therefore subjected to short-time tensile tests at temperatures ranging from 2500° to 4400° F.

MATERIALS, APPARATUS, AND PROCEDURE

Materials

Swaged 1/2-inch-diameter bars of commercially pure sintered tungsten supplied by five different producers were evaluated. The processing schedules for the various bars were those ordinarily used by the individual producers. No attempt was made to procure materials of identical chemical composition, grain size, or working history, since one of the purposes of the investigation was to determine the variability of high-temperature strength and ductility properties of commercial grades of tungsten. However, a nominal purity of 99.9 percent tungsten was specified.

The results of chemical analyses of samples from the as-received bars are shown in the following table:

Source	Composition, ppm by weight							
	Fe (a)	Mo (a)	Cr (a)	Si (a)	С (ъ)	0 ₂ (c)	N2	H ₂ (e)
А	210	180	40	30	4	25	15	1
В	280	230	70	90	5	43	37	-
C	260	250	100	30	47	35	39	3
D	340	350	120	50	24	4 5	38	2
E	160	390	50	30	26	35	2 8	2

^aSpectrographic analysis.

Density of the swaged bars was greater than 19.2 grams per cubic centimeter. Material A was the material evaluated in the previous study (ref. 1).

Tensile specimens of the type shown in figure 1 were machined from the 1/2-inch-diameter swaged bars and were evaluated in the as-received

^bConductometric analysis.

^cLeco oxygen determinator.

dMicrokjeldahl plus colorimetric using Nessler's reagent.

eCombustion method.

condition. Microstructures of the as-received bars are shown in figure 2.

Apparatus

The equipment and test procedures used were those described in reference 1, but some modifications were required to permit testing at temperatures above 3700° F. The 15-kilowatt, radio-frequency, induction heater used in the previous study was replaced by a 30-kilowatt motorgenerator set with a frequency of 9600 cycles per second. This required modification of the induction coil and the use of a heavier-walled susceptor tube. For this study, a seamless tantalum tube, 2 inches long by 3/4-inch outer diameter with a 0.060-inch-thick wall was heated by a two-turn induction coil. The tantalum susceptor was positioned and insulated from the coil in the manner previously described (ref. 1). For temperatures up to 3500° F, Inconel grips were used; for higher temperatures, grips were fabricated from a molybdenum plus 0.5-percent-titanium alloy. Figure 3 shows the arrangement of the strain column and the heater in the test chamber. A photograph of the high-temperature tensile test equipment is shown in figure 4.

Procedure

<u>Temperature measurement.</u> - As in the previous study, temperature was measured with a tungsten-molybdenum thermocouple spot-welded to the surface of the tensile specimen at its midpoint. Calibration of the thermocouple was extended from 3700° to 4500° F by placing the thermocouple in a black body enclosure and measuring its temperature with a calibrated optical pyrometer.

The test temperature reported in table I is the maximum temperature of the specimen. Because the specimen is gripped outside the heater, there is a longitudinal temperature gradient along it, as described in reference 1.

Test procedure. - All tests were conducted in a vacuum of less than 2 microns. The specimen was heated to the test temperature in about 15 minutes and held at temperature for an additional 15 minutes. It was then loaded to fracture at a constant crosshead speed of 1/16 inch per minute. A commercial, screw-driven, tensile-testing machine was used; the load-against-time curve was automatically recorded. Total-elongation measurements of the fractured specimens were made by fitting the fractured halves together and measuring the distance between two 15-mil wires that had been spot-welded to the specimen prior to testing 1/2 inch above and below its midpoint. Percent-elongation values were calculated (table I) assuming an effective gage length of 1 inch. These values are somewhat low since the actual gage length is less than 1 inch because of the longitudinal temperature gradient in the specimen.

RESULTS AND DISCUSSION

Comparison of tensile properties of tungsten from different producers. - The results of the high-temperature tensile tests are presented in table I and figure 5. The ductility values (figs. 5(b) and (c)) indicate that a decrease in ductility at high temperatures is characteristic of commercially pure, sintered, and swaged tungsten and not just of the material studied in reference 1. However, the ductility is still appreciable up to 4400° F. Reduction-in-area data for the different lots of tungsten (fig. 5(b)) were similar. All specimens fractured with localized necking and high ductility (85 to 95 percent reduction of area) at 2500° F, but without localized necking and with relatively low ductility (below 42 percent reduction of area) at temperatures above 3500° F. Elongation followed a slightly different pattern in that three of the four lots of material for which elongation measurements were made showed a maximum elongation at 3000° F and a fairly rapid decrease in the 3000° to 3800° F range. (Elongation measurements were not made on material A in the previous investigation (ref. 1).) Material from source E showed much less elongation at 3000° F and exhibited its maximum elongation at 3500° F (fig. 5(c)). Reasons for the change in the mode of fracture of tungsten at high temperatures are not known, and work aimed at understanding this phenomenon is continuing.

The curve of tensile strength against temperature (fig. 5(a)) for material from source E differed from those for materials from the other sources. The tensile strength at 2500° F for the E material was considerably lower than that of the others; however, at 3000° F, this material was stronger than the others. This suggests that the tungsten from source E was less severely strain-hardened and thus had a higher recrystallization temperature than did the other materials. An alternate possibility is that the tungsten from source E may have been "doped" with additives, which could influence the high-temperature strength by their effects on recrystallization and grain growth. Such additives are commonly added to tungsten intended for lamp-filament use and are known to influence grain structure at high temperatures. Since these additives may volatilize during the sintering process, the fact that they have been used is not usually revealed by chemical analysis. At temperatures above 3500° F (where the effects of strain-hardening would be expected to have been removed by annealing) material from all sources had about the same strengths. The average values for the specimens evaluated were 10,600 at 3500° , 6500 at 4075° , and 4400 psi at 4400° F.

Metallographic examination of fractured specimens. - Portions of all the fractured specimens were polished and examined metallographically to determine whether there were differences in recrystallization, grain growth, or deformation behavior of tungsten from the various sources. These examinations revealed no apparent reason for the different tensile properties of material from source E in the 2500° to 3500° F temperature range.

There were, however, some differences among the materials that may be significant. For example, materials from different sources exhibited widely differing amounts of porosity in the fracture region after tests at 2500°F. Material from source B had almost no porosity in the fracture zone, while material from source D showed considerable porosity (voids or cracks, fig. 6). Material from the other sources exhibited an intermediate amount of porosity. This porosity in the fracture zone apparently did not seriously affect the tensile strength of the tungsten from source D, for it was only slightly less strong at 2500°F than material from sources that showed less porosity.

Metallographic examination also revealed that there were significant differences in grain size of the materials at temperatures above 3500° F. There did not, however, appear to be any correlation between grain size and high-temperature strength or ductility. For example, figure 7 compares the microstructures at 3750° F of the coarsest grained material (E) with that of the finest grained material (B). Despite the relatively large difference in grain size of these specimens, the strengths were not greatly different (8140 psi for material from source B compared with 7650 psi for material from source E). Similarly, there was little difference in the fracture ductilities of these materials (25 percent reduction in area for the B material and 26 percent reduction in area for that from source E).

SUMMARY OF RESULTS

The following results were obtained from an investigation of the high-temperature tensile properties of commercially pure, sintered, and swaged tungsten:

- 1. The tensile strength of tungsten decreases relatively slowly in the 3500° to 4400° F temperature range. Average values of tensile strength for the specimens evaluated were 10,600 at 3500° , 6500 at 4075° , and 4400 psi at 4400° F.
- 2. The decrease in ductility of tungsten with increasing temperature above 2500° F, as measured by reduction in area at fracture in tensile tests, appears to be characteristic of commercially pure sintered tungsten. Material from five different producers behaved similarly in this respect.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, July 13, 1959

1. Hall, Robert W. and Sikora, Paul F.: Tensile Properties of Molydenum and Tungsten from 2500° to 3700° F. NASA MEMO 3-9-59E, 1959.

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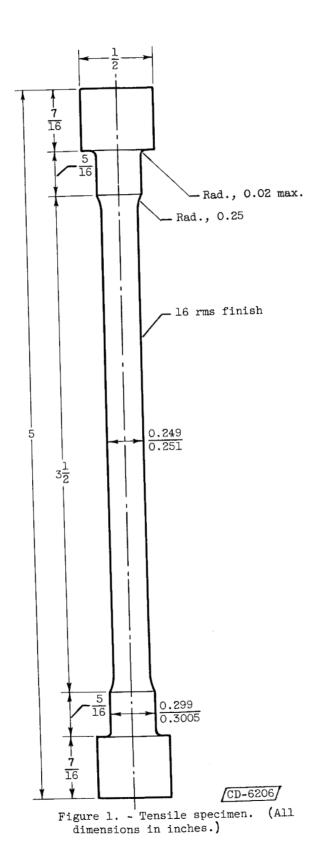
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TABLE I. - SHORT-TIME TENSILE PROPERTIES OF SWAGED TUNGSTEN
FROM FIVE COMMERCIAL PRODUCERS

Test temperature, OF	Source	Ultimate tensile strength, psi	Elongation in 1-in. gage length, percent	Reduction in area, percent					
2500	A ^a B C D E	49,350 52,200 45,400 50,600 37,800	25 26 25 25 25	95 94 94 86 87					
3000	A ^a B C D E	21,200 21,000 20,250 29,900 29,500	 46 47 51 22 20	73 78 81 79 79					
3120	A ^a	15,220		78					
3500	A ^a B C D E	10,130 11,500 10,850 11,250 9,180	23 32 25 35	34 28 41 34 42					
3750	A B C D	8,380 8,140 8,140 8,590 7,650	21 27 15 23	31 25 35 20 26					
4075	A B C D	6,080 6,810 6,890 6,840 5,820	 18 21 21 15	38 23 25 24 16					
4400 A B C D E		4,260 4,080 4,710 4,580 4,590	23 22 15	36 30 26 22 16					

^aData from ref. 1.



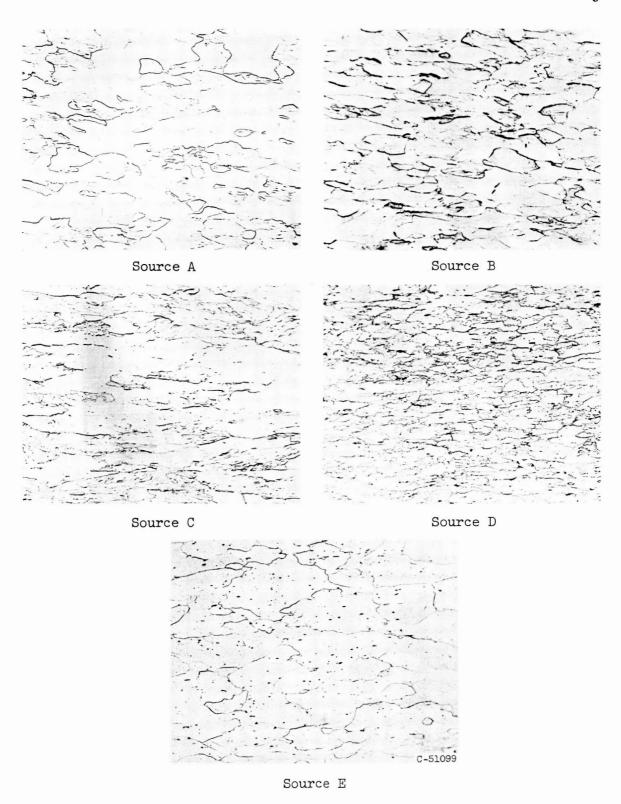


Figure 2. - Microstructure of as-received tungsten from five producers. Longitudinal section. X250.

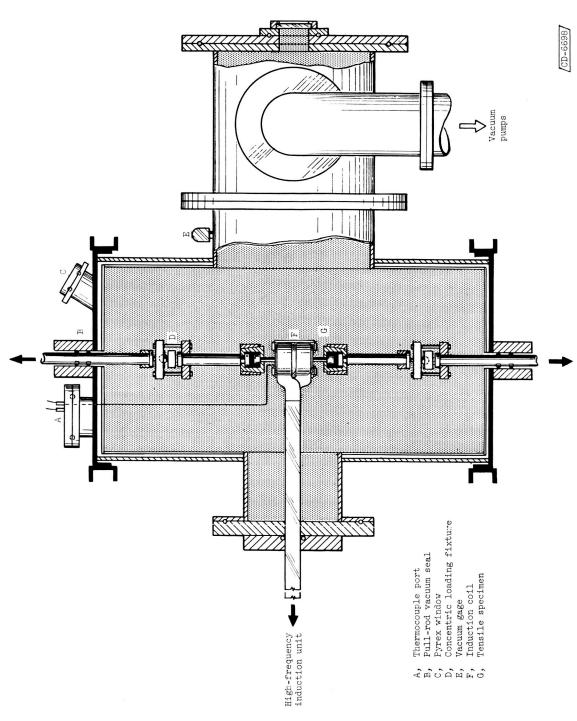


Figure 3. - High-temperature tensile test chamber.

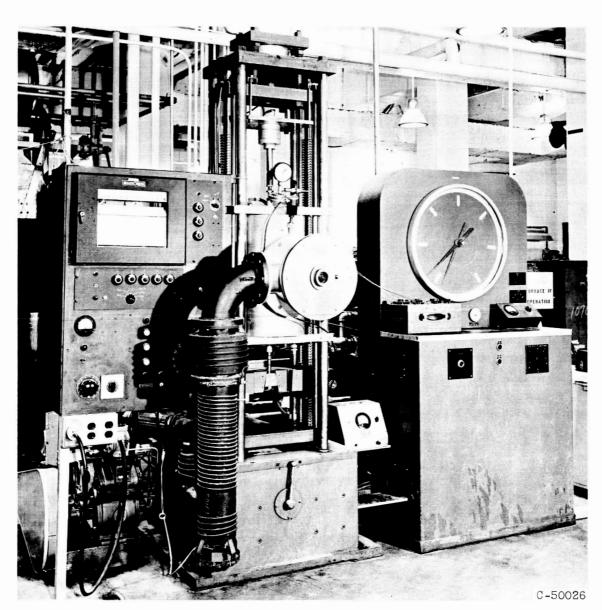
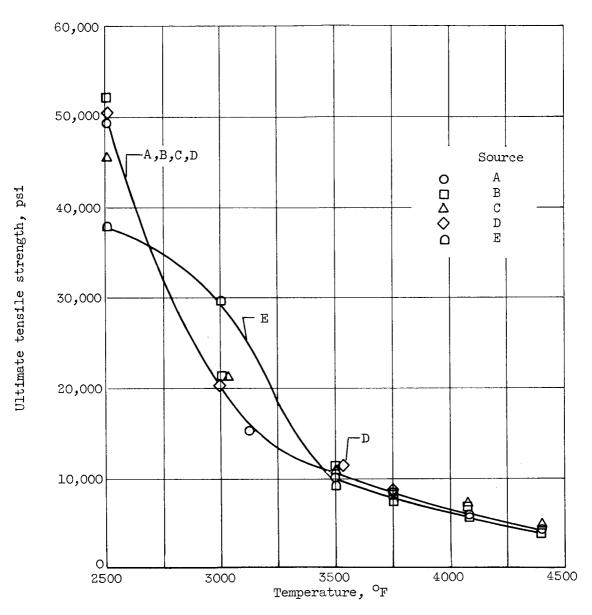
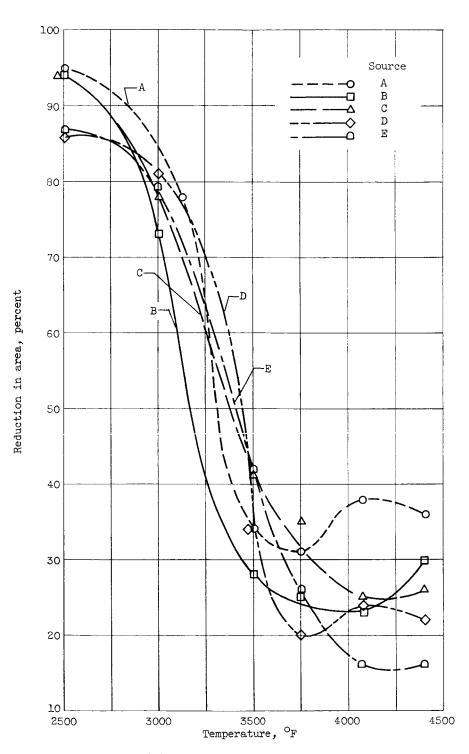


Figure 4. - High-temperature tensile test equipment.



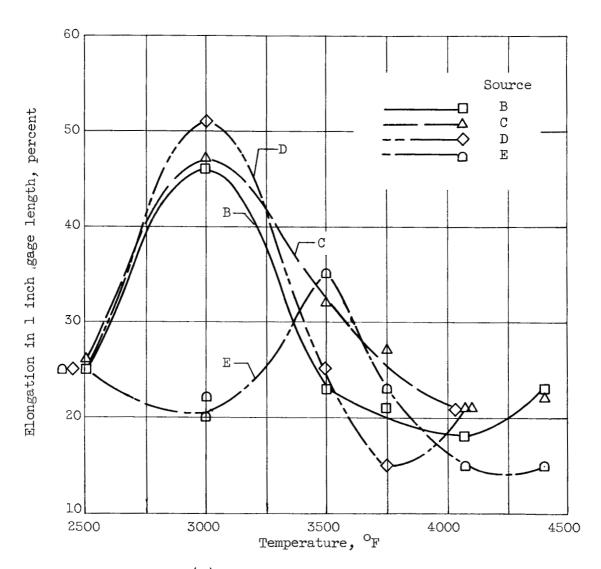
(a) Tensile strength.

Figure 5. - Effect of temperature on various properties of as-swaged tungsten from five different sources in short-time tensile tests.



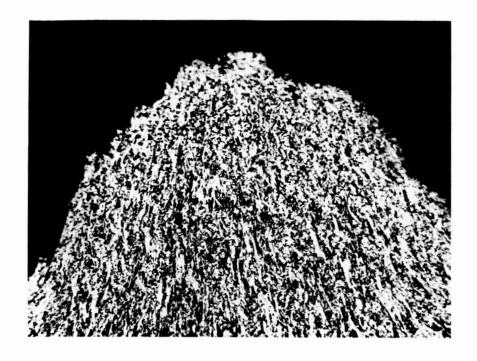
(b) Reduction in area at fracture.

Figure 5. - Continued. Effect of temperature on various properties of as-swaged tungsten from five different sources in short-time tensile tests.



(c) Total elongation at fracture.

Figure 5. - Concluded. Effect of temperature on various properties of as-swaged tungsten from five different sources in short-time tensile tests.



Source B

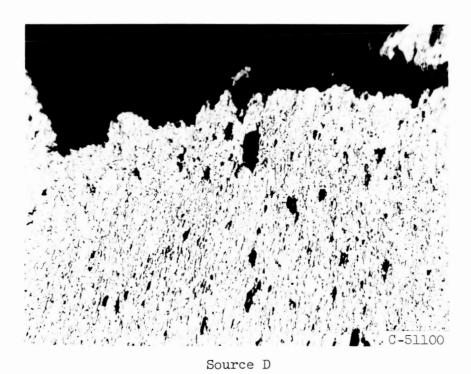
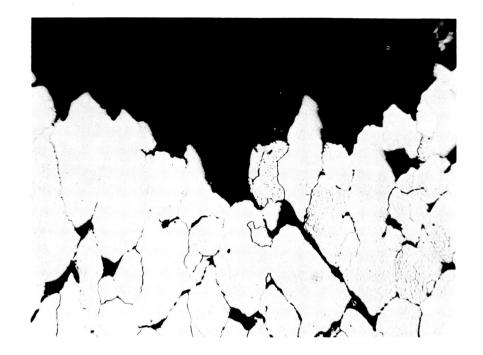


Figure 6. - Comparison of porosity at fracture face of materials from two sources after evaluation at 2500° F. X250.



Source B

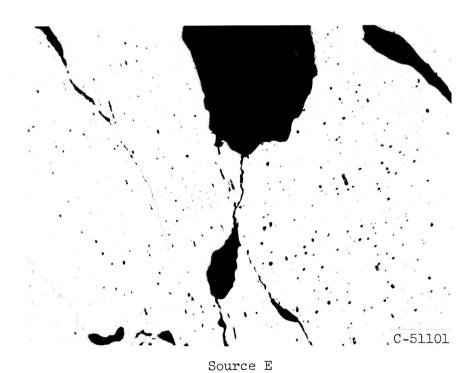


Figure 7. - Comparison of recrystallized grain size at fracture zone of materials from two sources after evaluation at 3750° F. X250.